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## Abstract

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The influence of thermal and mechanical treatments upon the elevated temperature stability, the microstructure and the mechanical properties of aluminum-aluminum oxide alloys is investigated. Compacts from atomized and flake aluminum powders were sintered at temperatures just below the melting point of aluminum in a vacuum of $10^{-4}$ Torr for several hours and were then hot extruded. During this treatment the hydrated oxide present on the surface of the powder particles decomposes, the water vapor produced reacts with the aluminum forming additional oxide, and the hydrogen developed is removed by diffusion to the surface. Extrusions of compacts so treated are stable at temperatures up to the melting point of aluminum and do not form blisters and internal defects typical of extrusions from untreated powder. Electron micrographs of extrusions from unsintered compacts show long parallel oriented platelets typical of the hydrated oxide skins on the original powder particles, while the oxide particles in extrusions from sintered compacts are broken up. The strength of extrusions from treated powder is somewhat lower, but their ductility is considerably improved over that of extrusions from untreated powder. The slip trace patterns on electron micrographs of extrusions which had been strained in tension before replication

indicated that the grainsize of the extruded alloys was approximately the same as that of the original powder particles. Coarse-grained alloys were produced by severe cold work and annealing of the extrusions. They have significantly lower room temperature strength, but about the same ductility as the vacuum sintered fine-grained alloys. The size and shape of oxides in the coarse-grained alloys is similar to those of the fine-grained alloys, but the particles are somewhat more uniformly distributed.

Author

## Introduction

In previous investigations [1,2] the properties and structure of aluminum alloys were described which had been produced by cold compacting aluminum powder and then hot pressing and extruding the compacts. Both the structure and the properties of these alloys differ significantly from those of commercially produced aluminum-aluminum oxide alloys. When commercial alloys are produced, they are given a sintering treatment in air at about 600°C between cold compacting and hot pressing [3]. During this treatment the hydrated oxide, which forms the coating on the aluminum powder particles from which the alloys are prepared is decomposed [4]. The chemically bound water which is released reacts with the aluminum and forms aluminum oxide and hydrogen. Most of the hydrogen is eliminated by diffusing to the interior or exterior surfaces of the compact, but it is difficult to eliminate all of it because the solubility of hydrogen in solid aluminum is very low even near its melting point. Extruded alloys which are not sintered before hot pressing and extrusion appear quite sound upon metallographic examination, but develop severe surface blistering and internal fissuring when they are heated to elevated temperatures after extrusion. Fissures and blisters have been observed even in commercial alloys which had been sintered in air, although they are much

less severe [5]. Because of the importance of this sintering treatment it was studied in this investigation in a modified form using vacuum sintering rather than sintering in air, to insure more complete elimination of occluded hydrogen. The technique is described and the effect of the treatment upon the structure of the alloys is documented.

It has been known for some time that extruded aluminum-aluminum oxide alloys are very finegrained, but an exact delineation of grains in electron micrographs has not been possible. In the conventional metallographic etching techniques etchants attack the interface between the second phase particles and the matrix in the alloys much faster than the grainboundaries. Grain boundaries can therefore not be distinguished in the etched structure. A surface deformation technique was developed by which the grains in the electron micrographs can be outlined. Typical examples of applying it to alloys are presented.

Although aluminum-aluminum oxide alloys in the extruded condition are quite finegrained, it is possible to produce exaggerated graingrowth in certain of these alloys having a relatively low oxide content by a sequence of cold working and annealing operations following extrusion. A discussion of the kinetics of grain coarsening in these alloys [6] and

preliminary data on the effect of the coarsening treatment upon tensile strength [7] have been published previously. The alloys to which the grain coarsening treatment was applied had not been sintered before hot pressing and extrusion and therefore contained internal cracks and fissures, which can be eliminated by including the vacuum sintering step in their preparation. The structure of sound coarse-grained alloys was compared with that of as-extruded alloys on the basis of electron micrographs of replicas. Finally, the tensile properties of alloys which had been vacuum sintered in the as extruded condition and of alloys which had been given the grain coarsening treatment after extrusion were compared with those previously reported [1,8] for alloys which had been cold compacted, hot pressed and extruded, omitting the vacuum sintering treatment.

Vacuum Sintering Treatment

The vacuum sintering treatment was developed for alloys prepared from five grades of aluminum powder, very similar to the grades used in previous investigations [1,2]. One of the powders was the atomized type; the other powders were flake type. The properties of these aluminum powders are given in Table I.

TABLE I

Properties of Aluminum Powders*

| Manufacturer | Grade Designation | Type | Particle Size or Flake Thickness, microns | Wt.Pct. Oxide |
|---|---|---|---|---|
| Reynolds Aluminum Co. | AT-400 | Atomized | 2 to 4, diameter | 2 |
| Metals Disintegrating Co. | MD-2100 | Flake | 0.8 | 3.0 |
| Metals Disintegrating Co. | MD-5100 | Flake | 0.4 | 5.7 |
| Metals Disintegrating Co. | MD-3100 | Flake | 0.4 | 9.3 |
| Metals Disintegrating Co. | MD-7100 | Flake | 0.17 | 12.3 |

(1)

* From Lenel, Backensto and Rose.

The flake powders, which are the type used in aluminum paint, were employed in this investigation so that the influence of the initial oxide distribution in the powder might be followed through the entire fabrication procedure. The shape and size of the powder particles is known, and the oxide can be assumed to be present only on the particle surface. These powders differ from the ones used in the production of commercial aluminum-aluminum oxide alloys, which consist of larger isometric particles, in which most of the oxide is embedded in the inside of the particles.

In order to produce sound extrusions from the flake powders, the lubricant remaining from the powder milling process was removed before compacting by first dissolving most of it in xylene, and then heating the powder in a vacuum of less than 50 microns to 450°C for 2 hours. After this treatment the powders were compacted under a pressure of 20,000 psi into compacts 1" in diameter. This pressure produced a compact with sufficient green strength for handling, but permitted the retention of a high degree of interconnecting porosity.

The compacts were then sintered in a vacuum of better than $10^{-4}$ Torr. The sintering temperature for all materials was 630°C. The compacts were heated to temperature under

vacuum. The time at temperature required for degassing varied according to the oxide content of the material.

After vacuum sintering, the compacts were hot pressed at 540°C for 20 minutes at a pressure of 20,000 tsi to raise the density to 2.70 gms/cc before extruding. They were then extruded using the indirect extrusion technique. The alloys with lowest oxide content, AT-400 and MD-2100, were extruded at 540°C. The alloys with higher oxide content required a temperature of 590°C to produce satisfactory extrusions.

In order to determine the adequacy of the vacuum sintering treatment, a test was developed in which the samples after extrusion were heated to 640°C in air for 5 hours. The high temperature for this test was chosen, because it was found that if the gas content was high enough to cause eventual blistering between 500°C and 600°C, blistering would occur in a very short time (less than one hour) when the material was heated to within several degrees of the solidus temperature (643°C).

After the heat treatment, a dark oxide film present on the specimens was etched off in dilute hydrofluoric acid. The specimens were then examined for surface blistering and in some cases sectioned and examined metallographically for

internal fissures. On the basis of this test, a vacuum sintering time of 10 hours is necessary to remove hydrogen from AT-400, MD-2100 and MD-5100 powder compacts. The sintering time was increased to 15 hours for MD-3100 powder compacts and MD-7100 powder compacts.

Effect of Vacuum Sintering upon Structure

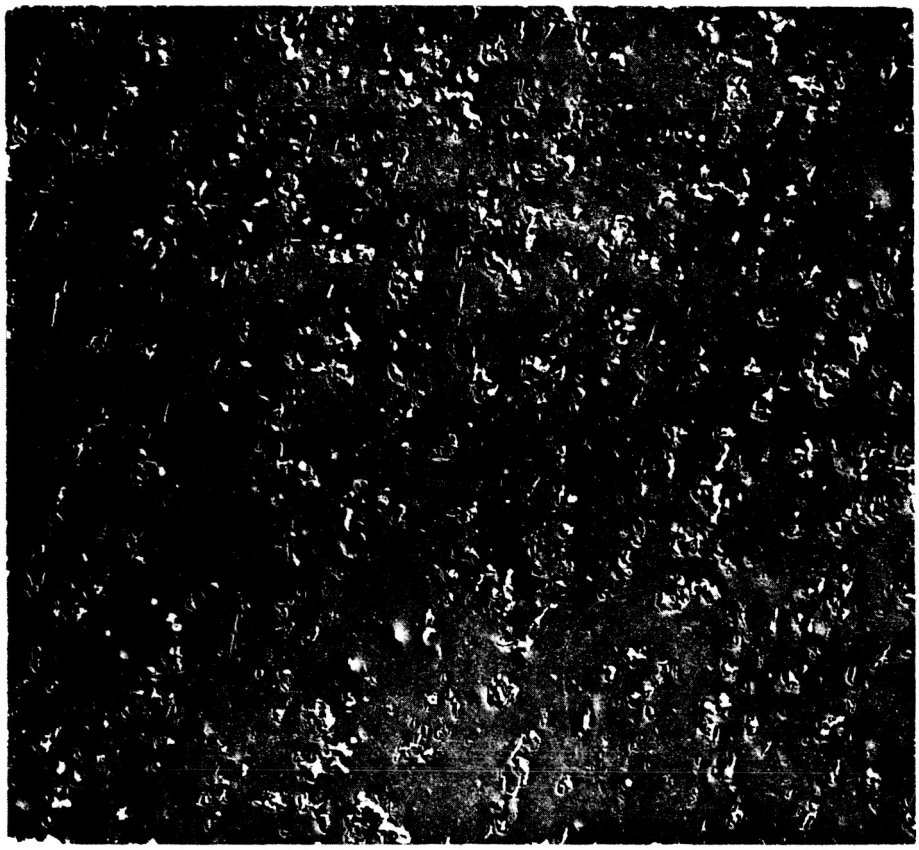
The effect of the vacuum sintering treatment upon the structure of the extruded alloys is shown in figures 1 and 2. They are electron micrographs of carbon replicas from alloy MD-5100 extrusions. The alloy of figure 1 was cold pressed and then hot pressed and extruded without any intermediate sintering either in air or vacuum, the alloy of figure 2 was prepared from the same powder, but the compact was vacuum sintered before hot pressing and extrusion. The oxide particles in figure 1 appear as parallel platelets, retaining the shape of the oxide coating on the original flake type particles from which the alloy was prepared. This typical morphology is lost when the compact is subjected to the vacuum-sintering treatment during which the hydrated oxide particles react with the aluminum matrix. It is evident that during the reaction, the particles of the oxide phase break up and fracture. An inspection of the micrograph of Figure 2 may also lead to

Figure 1 10,000X

Electron Micrograph of Carbon Replica of MD-5100 Alloy Fabricated by Cold pressing, Hotpressing and Extrusion without Intermediate Sintering Treatment

Figure 2 10,000X

Electron Micrograph of Carbon Replica of
MD-5100 Alloy Vacuum Sintered 10 Hours
at 630°C before Hotpressing and Extrusion



the conclusion that the oxide phase platelets have spheroidized. However, a study of the alloy by transmission electron micrograph [9] has revealed no evidence of spheroidized oxide particles, but instead indicates that the original thin platelet shape is retained. What appear to be spheroidized particles are groups of smaller, broken-up particles which are not individually resolved by the replication technique. In previous work, the spacing between oxide particles in the structure of the alloys as determined on electron micrographs of carbon replicas had been correlated with their yield strength and creep rate [10,11]. Even though this technique does not resolve the very small particles, it is believed that the spacing between larger particles and clumps of particles as indicated by the surface replication technique should still be the critical parameter in the relationship between structure and mechanical properties [9].

Delineation of grain structure in extruded alloys

In order to delineate the grain structure of the as-extruded alloys, a surface deformation technique was used. When a specimen which has been metallographically polished is deformed, the grain boundaries will show up as a result of differential deformation between adjacent grains. The slip

lines on the surface, which are also produced by the deformation, will usually terminate in the grain boundaries and thus further help to locate them.

The specimens used for this study were in the form of flat tensile specimens 1/16" thick, which were metallographically polished on one side by mechanical techniques and vacuum annealed at 600°C for two hours to remove the effects of cold working introduced during polishing. They were then electropolished, and finally strained in tension to the maximum amount of uniform elongation. The polished surfaces were then replicated for electron microscopy.

Figure 3 is a micrograph of an extruded AT-400 alloy specimen prepared in this manner. It can be seen that the grains are very elongated, undoubtedly in the direction of extrusion. The minor axes of the grains appear to be about 1 to 1½ microns in length, while the major axes may be as long as 10 to 15 microns. These grains, although elongated, contain approximately the same volumes as the original spherical powder particles. Therefore, no grain growth has occurred during the hot fabricating process. The oxide particles are not uniformly distributed through the grains but are segregated near the grain boundaries. These particles, which are the fragments of the oxide films originally formed on the aluminum powder particles, act to inhibit movement of the grain boundaries.

Figure 3 15,000 X

Electron Micrograph of Carbon Replica of
AT-400 Alloy, Strained 18% in Tension
after Extrusion

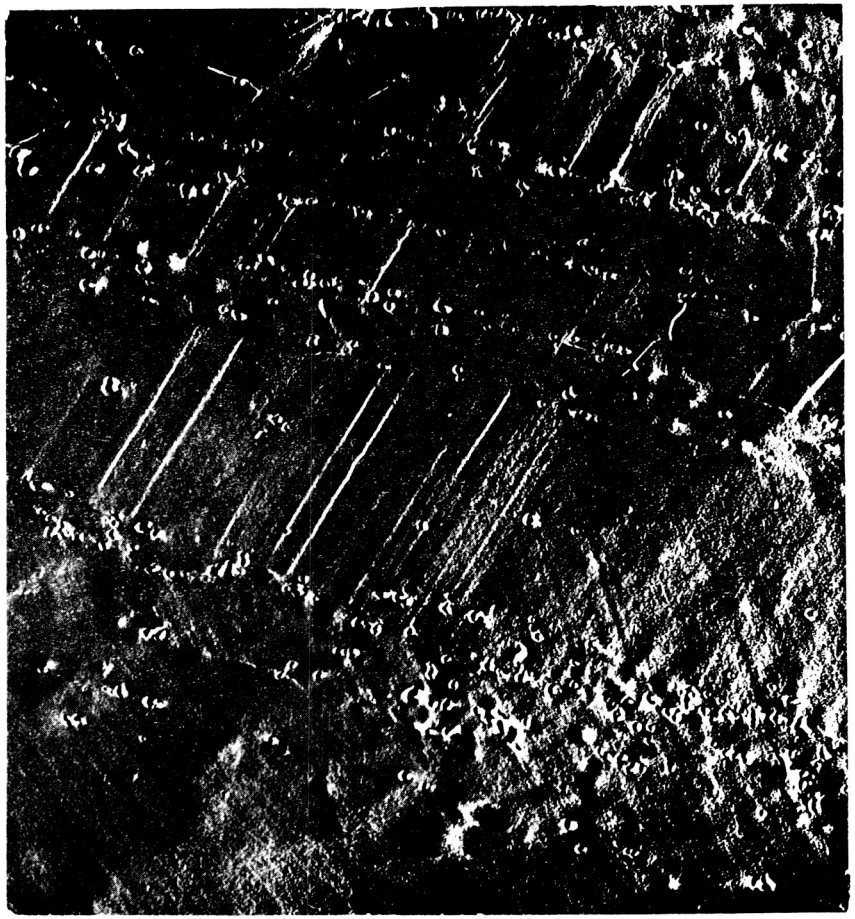


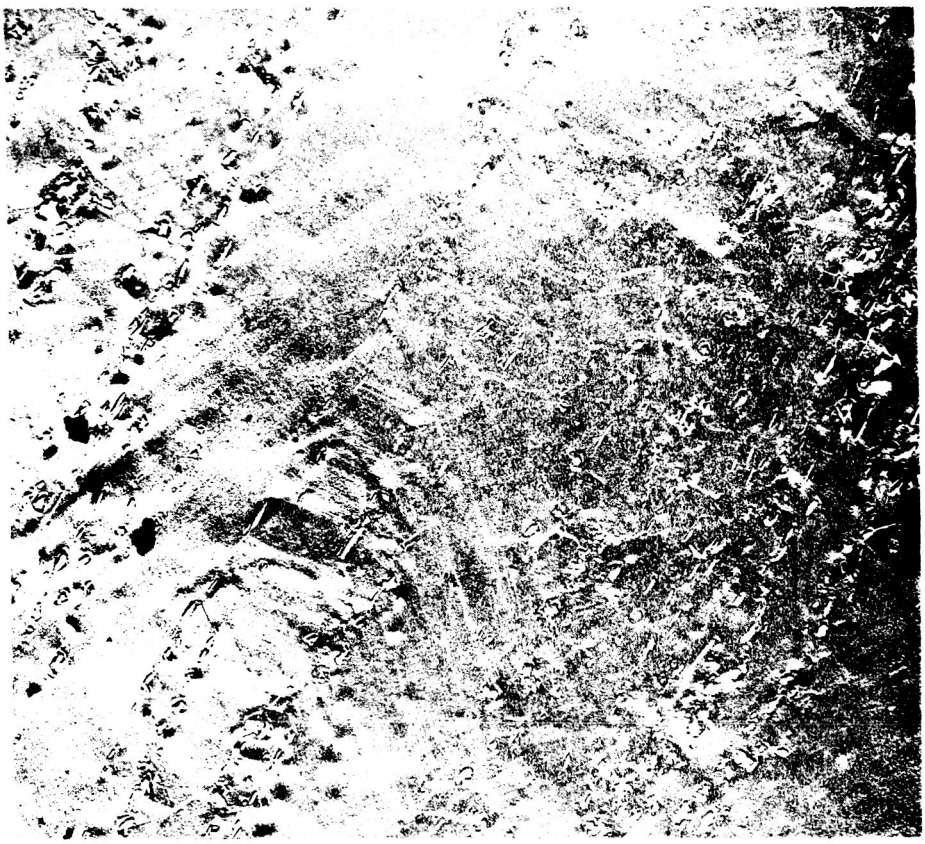
Figure 4 is a micrograph from a specimen of the extruded MD-2100 alloy. It appears similar to the micrograph from the AT-400 alloy except for the presence of more oxide particles. The grains, which are elongated, have minor axes of somewhat less than one micron. The MD-2100 powder is flake-shaped, rather than spherical, and the flakes are about 0.8 micron in thickness. Again no grain growth is indicated. This alloy also shows stringering of the oxide particles along the grain boundaries. In alloys with higher oxide contents than MD-2100 it is becoming more difficult to bring out the grain structure by the straining technique because of the increased resistance to deformation of these alloys. It is reasonable to assume, however, on the basis of the results from the other alloys, that no grain growth occurs in these materials during hot fabrication.

The grain coarsening treatment and its effect upon oxide particle distribution

A coarse grained structure was produced in the AT-400 and the MD-2100 alloys. Before being hot worked and extruded the alloys were given the vacuum sintering treatment in order to avoid the formation of blisters and fissures during later processing. After extrusion the alloys were cold worked by rolling in square and hexagonal grooved rolls and annealed in air in a resistance heated muffle furnace.

Figure 4 10,000 X

Electron Micrograph of Carbon Replica of
MD-2100 Alloy, Strained 13% in Tension
after Extrusion



To produce a completely coarse grained structure in two hours at 540°C, the AT-400 alloy required at least 70% reduction in area by rolling. The MD-2100 alloy required 85% reduction in area for grain coarsening under the same annealing conditions. An unsuccessful attempt was made to produce coarse grains in the MD-5100 alloy. A 95% reduction in area followd by annealing 10 hours at 643°C (the solidus temperature) produced no evidence of grain growth. Photomicrographs of the typical structure of grain coarsened AT-400 and MD 2100 alloys produced by oxidizing the alloy anodically and photographing it under polarized light have been published previously [6]. The grains are elongated in the direction of rolling with diameters of a few tenth of a millimeter in the direction normal to rolling and lengths up to several millimeters in the rolling direction. The increase in linear grainsize approaches therefore three orders of magnitude. The effect of the grain-coarsening treatment upon the distribution of the oxide particles is illustrated by comparing Figure 5, an electron micrograph of a replica from a coarse grained MD-2100 powder, with Figure 6 which shows the same alloy before the grain coarsening treatment. The severe cold reduction required to obtain a coarse grained material has produced a more uniform distribution of the oxide particles.

Figure 5 10,000 X

Electron Micrograph of Carbon Replica
of MD-2100 Alloy Grain Coarsened after
Extrusion by Rolling and Annealing

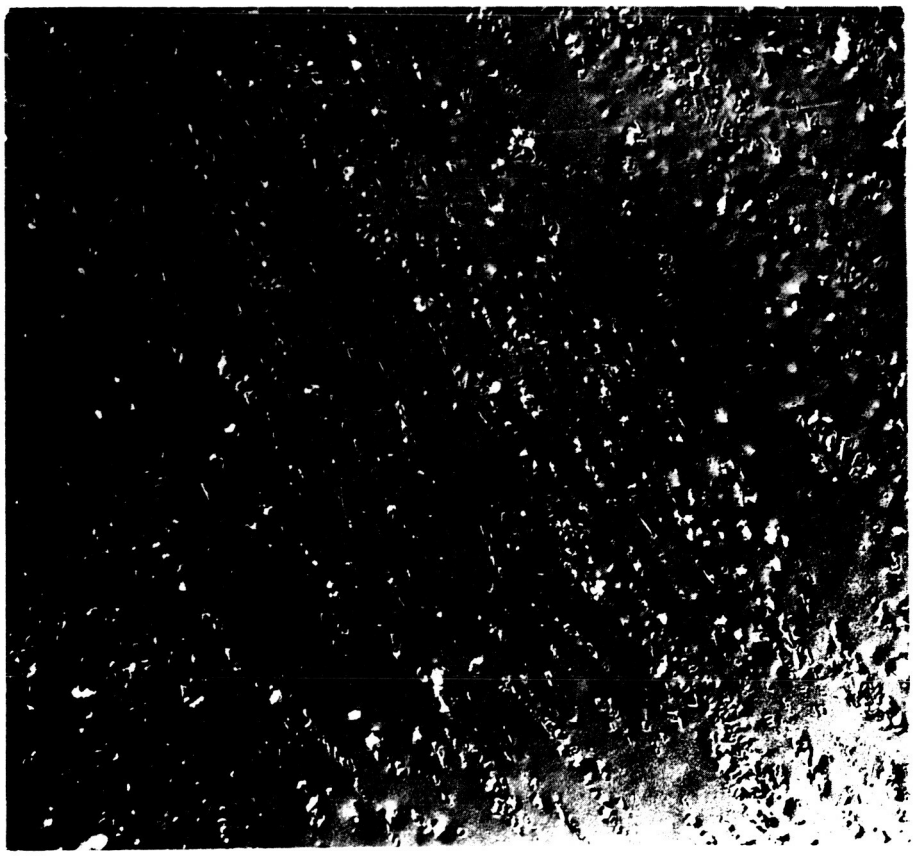
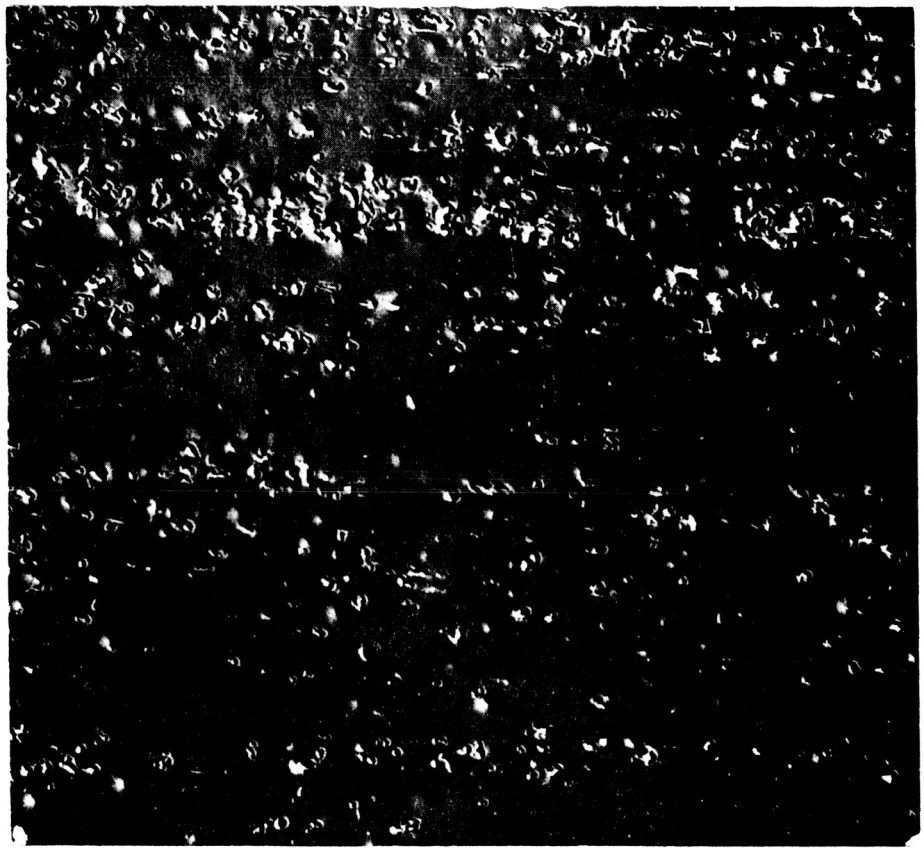


Figure 6 10,000 X

Electron Micrograph of Carbon Replica of
MD-2100 Alloy as Extruded



Room Temperature Tensile Properties

The room temperature tensile properties determined in this investigation include not only the usual 0.2% offset yield strength, but also the 0.01% offset yield strength. They indicate the yielding and the early work hardening behavior of the alloys. The stress-strain data required for this purpose were obtained in an Instron testing machine on standard proportioned reduced size tensile specimens with a 1/8" diameter gage section using electrical resistance type strain gages [12].

The results of tensile tests for vacuum sintered alloys and for alloys treated to produce a coarse grain size obtained in this investigation are combined in Table II with the data of Lenel, Backensto and Rose [1] and those of Lenel, Ansell and Bosch [8] on alloys in the extruded condition without a prior vacuum sintering treatment.

Vacuum sintering produces a loss in ultimate tensile strength in all of the alloys, and a similar loss in 0.2% offset yield strength for alloys from MD-2100 and MD-5100 powders, for which comparable values are available. Along with the decrease in strength, all of the vacuum sintered alloys show measurable increases in elongation. In view of the changes in the structure revealed by figures 1 and 2, the changes in mechanical properties are not surprising, since the effective particle spacing is significantly increased by the vacuum sintering treatment.

TABLE II

Comparison of Mechanical Properties of
Aluminum-Aluminum Oxide Alloys

| Material | Treatment | 0.01% Offset Yield Strength, | 0.2% Offset Yield Strength, | Ultimate Tensile Strength, | Elongation percent | Reference |
|---|---|---|---|---|---|---|
| AT-400 | V.S | 11,600 | 16,400 | 22,400 | 19.5 | |
| | C.W.+A | 9,300 | 13,300 | 17,400 | 13 | |
| MD-2100 | N.S | - | - | 35,100 | 14 | 1 |
| | N.S | - | 21,400 | 32,000 | - | 8 |
| | V.S | 11,400 | 19,700 | 28,900 | 16.5 | |
| | C.W.+A | 10,800 | 15,300 | 24,400 | 16.5 | |
| MD-5100 | N.S | - | - | 44,500 | 6 | 1 |
| | N.S | - | 30,400 | 46,400 | - | 8 |
| | V.S | 12,800 | 23,700 | 35,900 | 13.5 | |
| MD-3100 | N.S | - | - | 57,000 | 4 | 1 |
| | V.S | 19,700 | 37,900 | 51,500 | 6 | |
| MD-7100 | N.S | - | - | 59,000 | 2 | 1 |
| | V.S | 20,300 | 43,100 | 56,800 | 8 | |

V.S = Vacuum sintered before hot pressing and extrusion
N.S = No sintering treatment between cold compacting and hot pressing and extrusion
C.W.+A = Cold Worked and Annealed to produce a coarse-grained structure

The alloys with the coarse grain structure have still lower yield strength and ultimate tensile strength but the same ductility as the vacuum sintered alloys. Their yield strengths are, however, well above those for an aluminum matrix of similar composition without the dispersed phase.

Acknowledgement

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